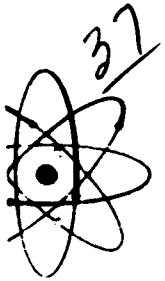

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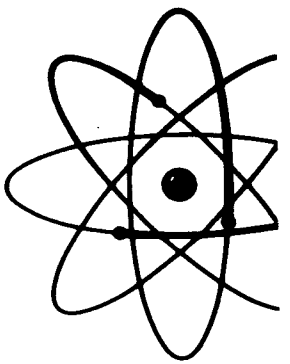

United States Atomic Energy Commission

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BERYLLIUM RESEARCH AND DEVELOPMENT PROGRAM

Quarterly Progress Report
Aeronautical Systems Division

for the Period

July 1, 1962 through September 30, 1962

S. H. Gelles

January 28, 1963

Nuclear Metals, Inc.
Concord, Massachusetts

Contract No. AF33(616)-7065
Task Nos. 73518 and 735104

This report has been reviewed
and is approved

A. R. Kaufmann
Technical Director

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF FIGURES

I. INTRODUCTION

This report describes the progress on the Beryllium Research and Development Program for the period July 1, 1962 through September 30, 1962. The progress for the previous periods is summarized in the final report covering the work accomplished from April 1, 1960 through September 30, 1961, ASD-TDR-62-509 (MMI-9516, in press) and in the quarterly reports MMI-9517, MMI-9519 and MMI-9522.

The following is a list of the projects for the continued program and the sites at which they are being carried out.

A Study of the Brittle Behavior of Beryllium by means of Transmission Electron Microscopy	Franklin Institute	F. Wilhelm H. G. F. Wilsdorf
Metallurgical Factors Affecting the Ductile-Brittle Transition in Beryllium	Lockheed Missiles and Space Company	M. I. Jacobson E. E. Underwood
Preparation of Ultra-Fine Beryllium Powder	National Research Corporation	P. L. Raymond P. J. Clough
Preparation and Evaluation of Fine-Grained Beryllium	New England Materials Laboratory	A. S. Bufford R. Widmer N. J. Grant
Recrystallization and Grain Growth in Beryllium	Pechiney Company	A. Saulnier R. Syre P. Vachet
Identification of Inclusions and Precipitates in Beryllium	Pechiney Company	A. Saulnier R. Syre P. Vachet
Preparation and Evaluation of High-Purity Beryllium	Nuclear Metals, Inc.	E. Levine J. P. Pemsler
Fabrication and Evaluation of Fine-Grained Beryllium Produced from Ultra-Fine Powders	Nuclear Metals, Inc.	A. K. Wolff

During this report period, visits were made to National Research Corporation and New England Materials Laboratory to review the programs being carried out at each of these sites. In addition, personnel from Pechiney visited Nuclear Metals, Inc., to discuss the programs on

recrystallization and grain growth in beryllium and identification of inclusions and precipitates in beryllium. A review meeting was held at Wright Field at which representatives from all of the subcontractor sites were present.

II. PROGRESS AT SUBCONTRACTOR SITES

A. Subcontract No. 4 - The Franklin Institute - F. Wilhelm and H. G. F. Wilsdorf - A Study of the Brittle Behavior of Beryllium by Means of Transmission Electron Microscopy

1. Introduction

Additional studies have been performed during this quarter on beryllium single crystals of commercial purity. In order to investigate the possible influence of vacancies on the dislocation behavior in contrast to the effect of impurities on dislocations, specimens were quenched and then deformed by compression.

2. Experimental Details

Small specimens with dimensions 0.8 by 6 by 25 mm were heated to 1150°C in vacuum in the rapid-quenching apparatus previously described[1]. The specimens were held at temperature for 30 minutes and then quenched by permitting ice-brine to rush into the apparatus, automatically stopping the induction heating. The quenched specimens were deformed by compression with a load of 100,000 psi., the compression axis being at an angle of 45° with the $[11\bar{2}0]$ direccion. A relative reduction in specimen thickness of approximately 10% was measured.

The specimens after thinning were investigated in the electron microscope. Fig. 1 shows the crystallographic orientation and the direction of viewing. Figures 2 and 3 give examples of micrographs obtained in this series. Noteworthy is a large number of rather irregular loops and spiral-shaped dislocations, with spiral diameters of 0.2μ and larger. Their mode of formation is yet to be studied. One specimen of the same orientation, receiving the same treatment, revealed a rather dense network of dislocations (Fig. 4). The orientation of

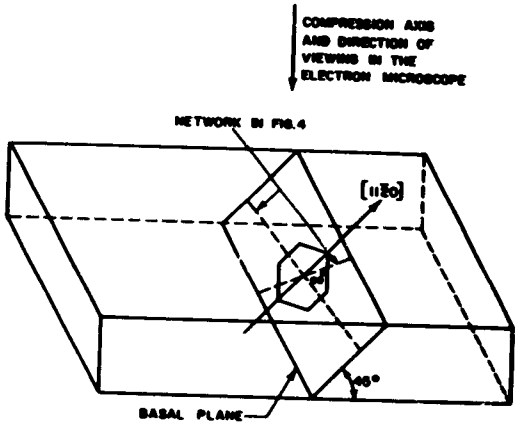


FIG. 1. CRYSTALLOGRAPHIC ORIENTATION OF BERYLLIUM SPECIMENS

Fig. 2 - Typical dislocation pattern with spirals in quenched and deformed beryllium. 40,000X.

Fig. 3 - Loops in quenched and deformed beryllium specimen. 40,000X

Fig. 4 - Dislocation network in a quenched and deformed beryllium single crystal specimen. 40,000X

the two sets of dislocations creating this network are found to form an angle of 60° with each other, each lying within 10° of $[10\bar{1}0]$ directions, as indicated in Fig. 1.

From micrographs of very thin specimens it was concluded, on the basis of dislocation contrast with respect to extinction contours, that approximately equal numbers of dislocations with positive and negative sign are present in these specimens.

In recent years the presence of long narrow dislocation loops has been observed in a number of deformed crystals. In fcc metals these loops lie parallel to $< 112 >$ and trail behind screw dislocations. Their long portions, therefore, have edge character and are of opposite sign. Various mechanisms have been proposed to explain the formation of the "dipoles", as these long narrow loops are called. In our opinion, dislocation dipoles are due to the interaction of point defects with glide dislocations and may provide significant information on the plastic behavior of beryllium.

It is, therefore, of interest to study the spectrum of widths of dislocation dipoles. Loops of widths less than 100 Å have not been found too frequently in the present investigation. Some micrographs that have been taken of polycrystalline vacuum-melted Pechiney flake specimens (as received) are most suitable for this purpose and show a sufficient number of loops to allow a meaningful evaluation. (See, for example, Fig. 18 of Ref. 2). In appraising this evaluation, it should be remembered that the crystallographic orientation of the individual crystal grains was not known. Some loops may therefore appear in projection. This will not affect the conclusion appreciably, however, because a number of very long, but extremely narrow loops leave little doubt of their dipole character. Loops that are listed as having a width of 50 Å are not resolved in the micrographs. Their widths may actually be smaller, down to few atomic distances. They are recognized to be loops by their contrast, by their alignment with long resolved loops, and by occasional flared sections along their lenth. Narrow

Figure 18 - Microstructure of flat extruded from Brush QMV -200 mesh powder.

loops of extremely short length (< 100 Å) are not considered in this study. They may be of a different nature than longer dipoles. A small fraction of the loops counted are open loops; i.e. they are actually long, drawn-out double dislocations, as may be seen, for example, in Fig. 12 of Ref. 3. They are listed as "loops" when their length to width ratio exceeds 5. Several micrographs have been evaluated in this way, representing a total area of $25\mu^2$, equivalent to a specimen volume of $5\mu^3$. A graphical representation of loop widths encountered is given in Fig. 5. The vertical markers represent the number of loops, of a given width, that were counted. To facilitate the interpretation of these values, the range of loop widths was divided into intervals of 100 Å. The number of loops counted which fall into the given interval is indicated by the broken line in Fig. 5. This histogram represents the density distribution of loops as a function of their widths. It is apparent that the number of loops with width < 50 Å increases sharply.

The dislocation density in the micrographs discussed is found to be 3×10^9 cm/cm^3. The contribution of the narrow dislocation dipoles with widths < 50 Å (and lengths up to 0.6μ) to the total dislocation density amounts to ≈40%, small short-length loops (width and length < 50 Å) contributing less than 2%.

3. Future Plans

Work has started on single crystal beryllium which has been reannealed after deformation in order to study polygonization networks in the dislocation structure.

MMI expects to prepare additional beryllium samples of such crystallographic orientations that dislocation patterns in prism planes can be observed after tensile deformation. These studies will permit the interpretation of observations made in earlier studies, which were concerned mainly with dislocation projected on the basal plane.

The investigation of dislocation dipoles and the detailed nature of glide dislocations will continue.

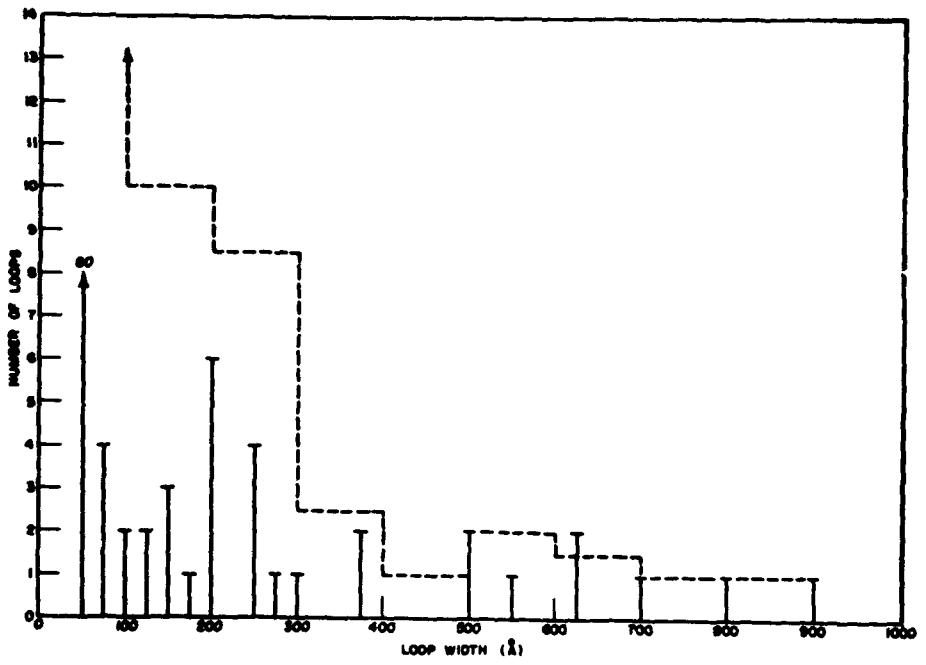


FIG 8. SPECTRUM OF LOOP WIDTHS

B. Subcontract No. 5 - Lockheed Missiles and Space Company - M. I. Jacobson and E. E. Underwood - Metallurgical Factors Affecting the Ductile-Brittle Transition in Beryllium

1. Material Acquisition

All of the beryllium necessary to conduct this investigation was received during the present quarter. The requisite hot pressings and extrusions were fabricated by Nuclear Metals from powders supplied by Brush Beryllium and Pechiney. Each company supplied three mesh sizes of powder, all attritioned from the same starting material so that variations in composition between different mesh sizes would be kept to a minimum. However, because the smaller mesh sizes required longer grinding times and/or impurities tend to segregate with the fines, some differences in composition were unavoidable.

The powder supplied by Brush had the following chemical analysis:

Analysis of Brush Beryllium Co. Powder

(ppm by weight)

	-60 + 80 mesh	-150 + 200 mesh	-325 mesh
Al	500	600	1300
C	800	900	800
Fe	1300	1500	1500
Mg	200	200	200
Si	400	400	400
BeO	0.41%	0.57%	1.40%

One hot pressing, 4-inch dia. by 3 inches, and one extruded flat, 3 inch wide by 1/2 inch thick by 20 inches long (approx.) were fabricated from each of the above mesh sizes.

The analysis of the Pechiney CR grade powder was as follows:

Analysis of Pechiney CR Powder

(ppm by weight)

	-50 + 120 mesh	-120 + 200 mesh	-200 + 350 mesh
Al	70	100	190
C	260	210	280
Fe	155	195	300
Mg	10	10	10
Si	152	205	230
Ca	100	90	75
Cu	60	90	35
BeO	0.42%	0.68%	1.17%

Again, one hot pressing and one extrusion were prepared from each of the mesh sizes.

Although grain size is the primary variable between each of the pressings and extrusions, it is quite apparent from the above analysis that there are significant compositional variations, particularly in regard to iron, aluminum, and BeO, between the mesh sizes of each type of powder. Although these will be taken into account in the final analysis of the data, it will nevertheless be very difficult, if not impossible, to distinguish between effects due purely to grain size or purely to composition within each powder grade. However, as the Pechiney material is an order of magnitude purer than the Brush material, it should be possible to attribute differences in mechanical properties of the two materials to differences in composition.

2. Experimental Work

The hot pressings have been sent to the beryllium shop for machining into tensile specimens. Round bar specimens are being prepared, with the axis of the sample parallel to the direction of pressing. The extrusions will be machined after pole figure determinations have been completed. This work is currently in progress.

Metallographic samples are being prepared from all of the material so that grain sizes can be obtained and microstructural examinations made.

It appears from preliminary examinations that the grain shapes of extruded material cannot be characterized as simple elongated cylinders. In view of the extreme importance of "grain diameter" to the theoretical treatments of the ductile-brittle transition, a careful investigation will be made of the anisotropy of mean grain dimensions in three major directions. Details of the quantitative treatment of anisotropic grain shapes will be discussed in subsequent reports.

Although no results can be reported at the present time, it is anticipated that metallographic and x-ray examinations of the as-received materials will be completed during the next quarter, and that the tensile test program will be well under way.

In view of the fact that some $15,000 of the original contract appropriation is to be diverted to a new program, some revision of the present program will be necessary. One of the ways being considered to reduce the scope of the program is to test single samples rather than duplicates. While it is recognized that this is an undesirable procedure for beryllium, if testing is carefully performed, it should be possible to construct reliable ductile-brittle transition curves. In cases where extreme discrepancies exist, duplicate tests can be made. Also, consideration is being given to eliminating strain rate as a variable. These points will be discussed more fully in subsequent reports.

Some thought is being given to the possible role of deformation twins with regard to crack initiation. Although surface twins are generally held to contribute to brittleness in beryllium, this may not always be so with interior twins. The microstructures of tested specimens will be checked systematically to determine the relationship of twinning to crack origin and crack path. Such factors as twin density, shape, size, distribution, and intersection, if treated quantitatively, may prove important to this evaluation.

C. Subcontract No. 8 - National Research Corporation - P. L. Raymond and F. J. Clough - Preparation of Ultra-Fine Beryllium Powder

1. Introduction

The object of this program is to produce ultra-fine beryllium powder for use in further studies to produce fine-grain beryllium bodies. A second objective is to determine the optimum conditions for producing ultra-fine beryllium powder of the desired particle size distribution and highest purity.

2. Progress of Program to Date

a. Equipment

Construction of all components of the system was completed early in the present period. With the completion of the ventilation system it was possible to test the air handling equipment for design

velocities and air flow pattern. The interior of the large tank was painted white to improve visibility and to aid in maintaining a clean system.

The evaporator and drybox were first operated using aluminum as a source material to produce ultra-fine aluminum powder. This permitted the technicians to become familiar with all handling and operating procedures to be used when working with beryllium without the actual hazard of beryllium. A view of the evaporator exterior is shown in Fig. 6.

A detailed set of instructions and explanations was prepared for all personnel who will be working on the program.

b. Powder Preparation

At the completion of vacuum testing and familiarization runs with aluminum powder, a series of seven beryllium powder production runs was conducted to produce approximately 400 grams of powder for Nuclear Metals. This powder will be delivered as soon as the Nuclear Metals drybox has been modified to accommodate the powder containers and pressing die. It is expected that delivery of the first lot of powder will be made early in October.

The operating conditions for all powder production runs to date have been held essentially constant. Approximately 150 grams of vacuum-melted beryllium pebble was used per run. The evaporation source consists of a 3-inch OD graphite crucible coated with a slurry of boron nitride. This boron nitride coating is to prevent reaction between the graphite and the beryllium to form an insoluble carbide which would rapidly halt the evaporation if it were allowed to form. The crucible is heated by induction to approximately 1750°C and the evaporation cycle is carried out for 20 to 60 minutes.

The powder recovery efficiency of the seven runs ranged from a low of 27% of the beryllium evaporated to a high of 61%. Two runs were nonproductive owing to the formation of cracks in the evaporation crucible before evaporation was started. Additional work is necessary

[illegible] of operation of evaporation and glove box facility.

to improve the life of the evaporation source and also to develop a more consistantly reliable evaporation source.

The powder is packaged in the drybox adjacent to the evaporator. Several ounces of powder are placed in pint-sized jars and these are sealed with plastic tape. The jars are then sealed in steel shipping containers with rubber gaskets at the covers. Since all packaging is done under an inert argon atmosphere, a double seal is provided to maintain the highly reactive beryllium powder in its original state as it comes from the evaporator.

c. Equipment Modification

As a result of discussions with personnel from Nuclear Metals during a visit to view the NMI drybox, it was decided to incorporate an argon recirculating train with a titanium getter oven and cold trap with the NRC drybox and the beryllium evaporator. Qualitative tests indicate that an improvement in the purity of the argon atmosphere has resulted since the installation of the purifying train.

d. Air Sampling Tests

A series of air sampling tests was conducted by the industrial hygiene consultants while the system was being operated under varying conditions. All routine operations were conducted without permitting hazardous quantities of beryllium powder to escape to the atmosphere of the tank. The one operation that did require modification was that in which the main evaporator is washed down, base removed for cleaning and entry made into the tank itself. It was indicated that substantial amounts of beryllium powder could be released in this operation. Modifications therefore have been made to vent the evaporator with the exhaust system, before removing the base and before personnel enter the tank for clean-up purposes. During the course of the tests all personnel were fitted with full face respirators and protective disposable clothing so that no accidental inhalation of beryllium could occur.

3. Planned Operations

As soon as Nuclear Metals has had an opportunity to evaluate the initial lot of powder, additional material will be produced for subsequent studies. Meanwhile, a few selected powder production runs will be conducted in an effort to further improve the quality and increase the powder production yeild. It is also anticipated that small samples of powder will be sent out for electron microscope study in an effort to determine the particle size distribution of the powder.

B. Subcontract No. 9 - New England Materials Laboratory - A. E. Bafford, R. Widmer and W. J. Grant - Preparation and Evaluation of Fine-Grained Beryllium

1. Introduction

The objective of this work is to prepare beryllium powder of approximately 1μ particle size, to evaluate the mechanical properties of fine-grain products prepared from this powder and to determine the effect of oxide on these properties.

Initial work has been concerned with the various variables associated with the grinding of beryllium in an attritor. Of particular concern is the beryllium particle size and impurity content. Previous work[4] in a small attritor utilizing methyl alcohol as the grinding fluid with -200 mesh Brush QMV powder indicated high iron contents after 12 hours' grinding to the 1μ size range.

2. Experimental Results

A large stainless steel attritor was incorporated into an expanded drybox facility with improved procedures for maintaining pure inert atmospheres. It was expected that the large attritor would reduce the grinding time necessary to achieve a fine particle size and therefore reduce the level of iron contamination. Table I compares the operating characteristics of both attritors.

TABLE I

Comparison of Attritor Operating Variables

	Small Attritor	Large Attritor
Capacity (cc)	400	2500
Ball Charge, 3/16-inch diameter (gm)	1500	15000
Liquid Charge (ml)	150	1500
Powder Charge (gm)	150	350-400
Speed (rpm)	250-360	250-300

The first experiment in the large attritor utilized methyl alcohol as the grinding fluid with -200 mesh QMV beryllium powder. After approximately one hour of grinding, water necessary for the cooling of the attritor tank was accidentally stopped and the heat generated by the grinding action increased the temperature within the attritor to 140°F, causing evaporation of the alcohol and leaving a dried powder. Particle size analysis by metallographic techniques indicated an average size of less than 5μ, and chemical analysis of 300°F vacuum-degassed powder performed at MMI indicated 8.0 w/o BeO and 0.27 w/o Fe. The evaporation of the alcohol during this experiment precludes any valid comparison with other grinding studies. Since the original particle shape and oxygen content of the beryllium may affect grinding efficiency and contamination by minimizing abrasive action on the stainless steel, -110 mesh Pechiney beryllium powder was also ground in the large attritor with methyl alcohol as the grinding fluid. The results of the grinding action are shown in Table II where they are compared to those attained by grinding -200 mesh QMV Brush Beryllium powder in the small attritor[4].

It may be seen from these results that, for a given grinding time, the iron contents of the Pechiney beryllium ground in the large attritor are lower than those attained on the QMV material ground in the small attritor. The particle sizes that one attains in a given length of time appeared to be smaller for the grinding of the Pechiney in the large attritor. However, definite conclusion regarding this will have to await a direct comparison between the sizes as measured metallographically and those measured by the Fisher technique.

TABLE II

Iron Contents for GMV -200 mesh Beryllium Powder Ground in the Small Attritor Compared to those for Pechiney -110 mesh Beryllium Powder Ground in the Large Attritor. Methyl Alcohol Grinding Medium

Grinding Time (hrs.)	GMV (Small Attritor)		Pechiney (Large Attritor)	
	Size (μ) metallographic	w/o Fe	Size (μ) Fisher	w/o Fe
0	-74	0.05	10.4	0.048
1	-20		3.9	1.430
2	-15	2.46	3.1	2.060
3	-10		2.7	2.800
4	- 8	6.25	2.4	3.510

E. Subcontract No. 10A - Pechiney - A. Saulnier, R. Syre and P. Vachet - Recrystallization and Grain Growth in Beryllium

1. Introduction

During this report period, work at the Pechiney beryllium mill has been interrupted by a three-week vacation.

Fabrication of Pechiney beryllium SR flake has been continued and fabrication of the Brush beryllium which was received at the end of July has been started.

2. SR Grade Pechiney Beryllium

Three square plates, approximately 15 mm thick have been machined from the slabs upset from 1 cast billet and 2 sintered billets.

The plate dimensions are as follows: H-1190 SR, 147 x 147 mm; A-19 SR, 123 x 121 mm; A-18 SR, 108 x 109 mm.

The plates were macroscopically and radiographically examined after machining.

a. Macroscopic Examination

Macroscopic observations have been carried out satisfactorily by a 10-minute immersion in a bath of 5% sulfuric acid, 1% hydrofluoric acid and distilled water followed by copious rinsing.

Measurements show the following grain sizes: 0.5 to 3 mm for H-1190 (cast and forged) 0.05 to 0.5 mm for A-19 (sintered and forged from -50 +110 mesh powder) and 0.01 to 0.08 mm for A-18 (sintered and forged from -200 mesh powder).

The grain size is relatively homogeneous with the exception of the cast metal. Here the cast structure is still clearly visible on one of the faces of the plate (corresponding to the top side of the ingot where the solidification is clearly inward).

b. Radiography

Radiography has been carried out under conditions that would provide the best sensitivity taking into account the thickness of the plates. Kodak Type-M film was used with a voltage of 20-24 kv,

a current of 8 milliamps, an exposure time of 2 minutes at a distance of 1.10 meters. Figure 7 shows prints obtained from the radiographs. H-1190 SR shows sound metal with neither inclusions nor cracks, and a uniform background mottled as a result of crystal growth. A-19 SR is homogeneous and has a fine grain size with a fine dispersion of absorbent inclusions a few tenths of a millimeter in size. A-18 SR has some inclusions, four of which are of abnormal size and have a leaf-like appearance which is invisible on the positive. It has been determined that these inclusions are not on the surface of the plate.

The origin of the inclusions in the SR-grade beryllium powder (fabricated in a mill entirely of beryllium) has been localized to either the turning operation or the screening and compacting operation. In the turning operation this may arise from the fracture of the tungsten carbide tools in spite of the magnetic separation of the ground chips. In the screening or compaction of the powders, the inclusions arise from either the screens (stainless steel, bronze wires) or the mild steel can used during the compression. Effort will be made to isolate these inclusions in the hot-rolled sheet and to determine their chemical nature.

c. Mechanical Properties

Use has been made of peripheral pieces of the forging slabs for micrographic examination and for determination of the mechanical properties (hardness and tensile properties). Results of these examinations will be given in a later report.

d. Hot Rolling

Hot rolling conditions have been perfected on a hot-pressed plate and on two plates machined from a cast and forged ingot (material not from this program). The canning which has been successfully used during the entire rolling cycle is composed of a mild steel frame 15 to 20 mm wide fit to the dimensions of the beryllium plate (picture frame). Two mild steel covers of thickness equal to half that of the beryllium are are welded to the frame. A sheet of 1-1/2 mm thick stainless steel (18-8) is placed between each steel cover and the beryllium before welding.

(a) H-1190 SR

Fig. 7 - Radiographs of plates prepared from Pechiney SR beryllium.

(b) A-19 SR

(c) A-18 SR

Fig. 7, continued.

All of the sheets have been hot rolled (reduction 10:1) under the following conditions:

Rolling temperature: H-1190, 720-760°C; A-19, 750-780°C; A-18, 800-850°C.

Rolling speed: 25 m/min.

Reduction per pass: 20% between reheating, cross-rolling direction changed after each pass.

The final rolling steps require particular attention to avoid cracks caused by thermal constraints and to eliminate internal stresses. This is accomplished by reheating the sheets to 800°C, cutting approximately 50 mm off the edges, and slow cooling of the sheets in a container filled with fiberglass. Pickling of the mild steel in a nitric acid bath is then followed by peeling of the stainless steel.

Sheets of useable area averaging 350 by 350 mm have thus been obtained.

The sheets were then chemically etched in a nitric-hydrofluoric bath and radiographed. The etching of the slabs for rolling at low temperature is now in process. The cold working studies will be done during October.

3. Brush Beryllium

Chemical analysis of a sample of the Brush vacuum-melted beryllium lump has been performed. (Table III).

Three ingots 2-1/2 kg each have been melted under the following conditions: sintered beryllium oxide crucible; graphite mold 100 mm in diameter; melting under a vacuum of 10^{-4} mm Hg; holding and casting under a reduced pressure of argon at 1400°C; cooling in vacuum.

The ingots have been skimmed to a depth of 2 mm and then examined radiographically. The three ingots designated H-1329, H-1330 and H-1331 show large shrinkage cavities close to half the height of the billets, and cracks at the base of this cavity. Ingot H-1329 has been chosen for forging. The bottom part of this ingot has been cut to a height of 95 mm.

TABLE III

Analysis of Brush Vacuum-Melted Beryllium Lump

(Raw material and vacuum-melted ingots in parts per million by weight)

No.	History	Cr	Ca	Ni	Zn	Cu	Al	Si	Ti	Mg	Mn	Fe	B	Na	Mo	Cl
Lumps	As Rec'd	85	80	135	<80	80	950	400	-	230	80	1183	1	<100	<15	<20
H-1329	top	100	<30	125	"	70	760	380	150	65	"	1070	"	"	"	"
	bottom	90	"	120	"	54	"	"	90	75	78	1110	"	"	"	"
H-1330	top	105	110	"	"	45	"	"	240	30	82	1180	"	"	"	"
	bottom	"	<30	130	"	56	"	"	80	40	80	1070	"	"	"	"
H-1331	top	"	"	110	"	48	"	"	130	60	78	1155	"	"	"	"
	bottom	"	50	"	"	45	"	"	"	"	80	"	"	"	"	"

Chips have been taken for analysis from each of the ingots from both the top and the bottom. These results also are given in Table III.

Analysis of the ingots compared to the starting material shows a decrease in content of magnesium and aluminum.

The fabrication of powder from the Brush ingots is in process. The fabrication steps will be identical to those used on the Pechiney beryllium.

F. Subcontract No. 10B - Pechiney - A. Saulnier, R. Syre and P. Vachet - Identification of Impurities and Precipitates in Beryllium

1. Production and Fabrication of Alloys

a. Production of the Beryllium-Silicon Alloy

The difficulties in preparation of the 0.02 w/o silicon alloy from SR flake have been previously[4] reported. In a first attempt, ingot H-1231 did not appear homogeneous and had to be remelted. During the second melting, contamination by iron occurred. This probably came from the crucible (crucible is reserved for the fusion of SR beryllium alloys, and it was in this crucible that the beryllium-iron alloys had been made). Chemical analysis having confirmed this contamination, it was nevertheless decided to fabricate this ingot in order to examine the ternary alloy (beryllium-iron-silicon). A new Be-0.02 w/o Si ingot has been produced under the same conditions as used before. SR-grade flake beryllium from Lot 98 was used and the addition of pure silicon was in powder form. This billet is designated H-1332. Two samples have been taken from the top and bottom of the ingot after skinning. Two-thirds of the bottom part of the ingot can be used for extrusion.

Complete analyses of the cast alloys and of powder fabricated from two of them are shown in Table IV. The analyses for oxide done in the analytical laboratory of the fabrication facility (Calypso), performed by solution in bromine methanol, probably are high especially for the analysis of the powder. They can also be influenced by the presence of alloying elements.

TABLE IV

Analysis of SR Pechiney Beryllium Alloys

(.n parts per million by weight)

No.	History	Sample	Cr	Ca	Ni	Zn	Cu	Al	Si	Ti	Mg	Mn	Fe	Na	BeO	Cl	C
H-1226	BeFe cast	top	<10	<30	<25	<80	<5	<30	15	<25	<10	<5	2500	<100	1800	20	-
		bottom	"	"	"	"	"	"	<15	"	"	"	2400	200	2000	<20	145
	BeFe powder	-110 mesh	"	100	25	"	10	20	15	"	18	. 5	2015	<100	6800	-	-
H-1227	BeFe cast	top	<10	<30	<25	<80	<5	30	20	<25	<10	<5	1050	<100	2300	<20	-
		bottom	"	30	25	"	"	"	30	"	12	"	1150	"	2600	"	175
H-1228	BeFe cast	top	<10	<30	<25	<80	<3	30	<15	<25	<10	<5	500	<100	2600	<20	-
		bottom	"	"	"	80	"	"	"	"	"	"	500	"	2000	"	175
H-1229	BeAl cast	top	<10	<30	<25	<80	6	2200	30	<25	<10	<5	30	100	1800	<20	-
		bottom	"	"	"	"	<5	2100	<15	"	"	"	35	130	2000	"	210
		-110 mesh	"	70	20	"	18	2100	40	"	50	"	50	<100	20000	-	-
H-1230	BeAl cast	top	<10	<30	<25	<80	<5	300	20	<25	10	<5	45	100	1400	<20	-
		middle	"	80	19	<80	"	-	90	"	15	"	40	<100	1500	"	-
		bottom	"	<30	<25	"	5	500	220	"	<10	"	40	"	1600	"	180
H-1231	Be-Fe-Si cast	top	35	<30	35	<80	9	<30	200	<25	15	24	1740	<100	2000	<20	-
		bottom	20	90	25	"	5	55	200	"	"	19	1700	"	2300	"	200
H-1332	BeSi cast	top	<10	<30	10	<80	<5	30	300	<25	<10	<5	30	<100	2000	<20	220
		bottom	"	"	"	"	"	"	270	"	"	"	"	"	1800	"	210

b. Fabrication of alloys

(1) Extrusion. The beryllium-silicon alloys have been extruded under conditions as close as possible to those used for extrusion of the other alloys: canning in mild steel approximately 2 mm thick; hot pressing at 850°C under 100 kg/mm^2 pressure; extrusion into a flat under the conditions given in Table V; reduction in area of 9.5:1.

Two sintered billets, designated A-21 and A-20, have also been produced by grinding chips from ingots H-1226 (BeFe) and H-1229 (BeAl) respectively, cold pressing the -110 mesh portion, hot pressing in cans at 850°C at a pressure of 100 kg/mm^2, and extrusion into a flat under the conditions shown in Table V. The beryllium-aluminum extrusion proves to be very brittle, the cast and extruded billet being partially cracked and the sintered and extruded billet totally unuseable for rolling. The decrease of plasticity with increase in aluminum content has been reported elsewhere by Olds, et al.(5)

(2) Rolling. Three plates 60 by 50 mm have been machined from each extruded flat and canned in a sheet of 18-8 stainless steel welded on its sides. The rolling conditions adopted for all the alloys are as follows: major rolling direction, transverse to the extrusion direction; preheat temperature, 830-850°C; final rolling temperature, 780-800°C; rolling reduction, 12:1; 10% reduction per pass.

After removal of the can, the sheets were subjected to two 5% smoothing passes at a temperature of approximately 750°C, reducing the thickness to 1 ± 0.1 mm. The sheets were then etched in a HNO_3-HF bath. The sheets made by hot rolling the cast and extruded alloys exhibit a large grain size. The tendency toward cracking appears to be greater for the sheets made from the sintered and extruded billets (A-13 and A-21). The surface condition after etching is also worse (oxidation, inclusions) for the sintered sheets and for the alloys having large alloy additions (H-1226, H-1229, and H-1231 and A-21).

TABLE V

Beryllium Alloy Extrusion Conditions

Alloy	No.	Temperature °C		Extrusion Pressure Kg/mm2		Max. Speed m/mn	Appearance
		Billet	Container	Start	End		
Be-Fe-Si	H-1231	950	430	57	98	6.8	good
Be-Si	H-1332	980	450	30	73	1.3	good
Be-Fe	A-21	980	430	47	73	3.5	good
Be-Al	A-20	950	430	53	73	1.3	cracked

c. Analysis of the CR Pechiney Beryllium Sheets After Fabrication

Samples for analysis have been machined from each as-rolled sheet. The analyses obtained on sheets made from commercially pure flake are given in Table VI. It is seen that the iron, nickel and chromium contents of the powder and powder metallurgy sheet are clearly higher than in the cast ingot from which they come. This contamination, which originates from grinding in stainless steel, is all the more marked because the ratio of mass ground to grinding time was much less than in the normal technique of fabrication. This contamination is not seen on the beryllium-iron and beryllium-aluminum powders prepared from SR pellets because in these cases the laboratory mill used was made entirely of beryllium. The analytical results on the eight alloys produced will be presented in a later report.

d. Metallographic Examination of Commercially Pure Beryllium in the Sintered and Extruded State and in the Sintered, Extruded and Rolled State

Observations by optical and electron microscopy have been conducted on Pechiney CR grade beryllium (Billet A-13) in the two conditions, sintered and extruded, and sintered, extruded and rolled.

(1) Sintered and Extruded Metal

(a) Optical Microscopy. A relatively fine grain size has been observed on a section transverse to the extrusion direction (Fig. 8a). Numerous particles of oxide appear to be uniformly distributed. On the other hand, on a longitudinal section these oxide particles are clearly lined up along the extrusion direction (Fig. 8b).

(b) Electron Microscopy. These oxide particles (black spots) have been found on a carbon replica to be more or less in lines. (Fig. 9). Several electron diffraction patterns taken on an agglomeration of these black particles have clearly established these particles as beryllium oxide.

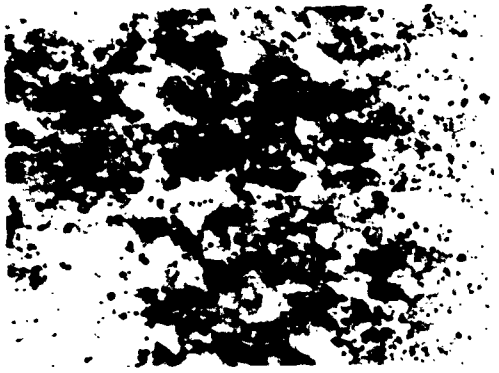
TABLE VI

Analysis of CR Grade Pechiney Beryllium Starting Flake, Billets, Powder and Sheet

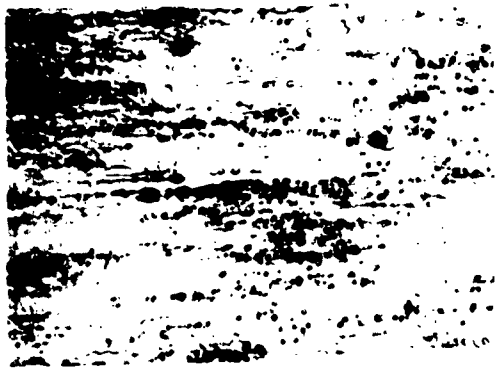
(in parts per million by weight)

No.	History	Cr	Ca	Ni	Zn	Cu	Al	Si	Ti	Mg	Mn	Fe	Na	BeO	Cl	C
Lot 4	Flake	<10	<30	45	<45	6	40	<15	<25	10	12	180	60	2700	320	<100
H-1168	Cast Ingot	6	<30	95	<80	48	40	<15	<25	<10	25	195	<100	360	<10	220
	Sheet	<10	100*	65	"	<5	45	80	"	35	12	230	350*	1200	-	-
A-13	-110 mesh powder	160	40	170	-	-	45	-	-	<10	-	1040	-	-	-	-
	Sheet	120	100*	140	<80	5	75	130	-	25	17	1100	250*	7400	-	-

*Possible contamination from Ca and Na by the solutionizing reagent.



(a) Perpendicular to extrusion direction.



(b) Parallel [illegible] direction

Fig. 8 - Copper [illegible] sintered and extruded [illegible]

Fig. 9 Commercially pure Pechiney beryllium sintered and extruded. Carbon replica taken parallel to the extrusion direction. 10,000X

(2) Sintered, Extruded and Rolled Metal
(thickness, 1 mm)

(a) Optical Microscopy. Fig. 10: The metal is heavily worked. Several particles of oxide are uniformly distributed.

(b) Electron Microscopy. Particles of oxide of several tenths of a micron have been found on a carbon replica. The sheet has also been thinned by chemical and electrolytic techniques for direct examination in the microscope. Particles of oxide have been found distributed in the interior of the grains and sub-grains without apparent relationship with the grain boundaries (Figs. 11 and 12). In certain places these particles have massed into rather large groups (not shown). Very few large inclusions have been observed and no precipitation is apparent even at high magnification (Fig. 12). It might be feared that the oxide particles would mask precipitation; however, these particles have a sufficiently characteristic appearance to be easily distinguished from eventual precipitates. In Fig. 11, it can be seen that dislocations are split. According to certain authors, this phenomenon occurs when the sample is oriented so that two Bragg reflections of simple indices are excited.

(3) Ultra Pure Beryllium. The observations which follow are reported for samples sent by Dr. Levine of Nuclear Metals in June, 1962 (second shipment), and are referenced as follows: Vacuum-melted CR grade Pechiney flake and vacuum-melted distilled beryllium.

(a) Radiographic Inspection. These two samples have been radiographed. Contamination on the edges observed on the first samples[6] were not seen on these samples. In the vacuum-melted CR Pechiney, numerous fissures were apparent which seem to follow the grain boundaries.

(b) Optical Microscopy. These samples appear to be very sensitive to working, because numerous twins were observed after polishing. They also contain a large amount of microporosity. There is a tendency toward the removal of grains during polishing.

Fig. 10 - Commercially pure Pechiney beryllium sintered, extruded and rolled. Sheet surface. 430X

Fig. 11 - Thin film of commercially pure [illegible] (A-13) sintered, extruded and rolled

Fig. 12 - Thin film of commercially pure beryllium, sintered, extruded and rolled. Film parallel to (0001). 50,000X

(c) Electron Microscopy. These examinations are only for the vacuum-melted Pechiney. Preparation has been a particularly delicate operation because of grain pull-out. On the preparations thinned chemically and then electrolytically, it has been possible to find several inclusions (Fig. 13) whose dimensions are of the order of 1 micron. The diffraction pattern given by one of these (Fig. 14 and 15) is rather poor. A large number of Kikuchi lines can be seen. This is evidence that the sample is too thick. Several spots can be seen having lattice spacings of the order of 3.1, 2.1 and 1.4 Å. These may correspond to either $FeBe_5$ (3.40, 2.08, 1.47 and 1.35 Å) or to silicon (3.14, 1.92 or 1.36 Å). The silicon appears more probable.

Mechanical thinning followed by electrolytic thinning has shown little advantage for the detection of inclusions. On the other hand, mechanical polishing introduces such a large number of dislocations that the appearance of the substructure completely changes (Figs. 16 and 17). On the sample thinned chemically, large dislocations and several elongated and oriented loops are observed.

III. WORK AT NUCLEAR METALS, INC.

A. Preparation and Evaluation of High Purity Beryllium - E. D. Levine and J. P. Pemsler.

1. Evaluation of Beryllium Powder Extrusions

During the present quarter, mechanical property evaluations was performed on extruded flats made from beryllium powder of various purity levels. As described in the previous quarterly report,[4] the flats were fabricated from Brush QMV -200 mesh powder, Pechiney SR -110 mesh powder, attritioned ingot Pechiney CR beryllium, attritioned 5-pass zone-refined CR grade Pechiney flake and attritioned double-distilled beryllium.

Microstructures of the as-extruded specimens are shown in Figs. 18-22. These photographs exhibit the influence of both initial particle size and purity on the extruded grain size and structure. For example,

Fig. 13 - Vacuum-melted CR grade Pechiney beryllium. Chemically and electrolytically thinned. 20,000X.

E.84.7

Fig. 14 - Vacuum-melted CR grade Pechiney beryllium Chemically and electrolytically thinned. 50,000X.

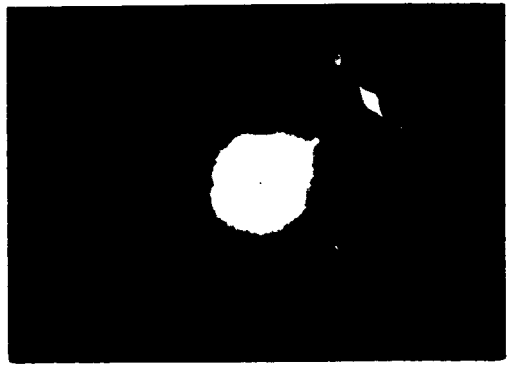
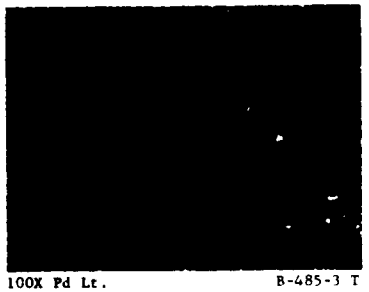


Fig. 15 - Electron diffraction pattern from area shown in Fig. 14.

Fig. 16 - Vacuum-melted CR grade Pechiney beryllium
Mechanically and electrolytically thinned
50,000X.

Fig. 17 - Vacuum-melted CR grade Pechiney beryllium.
Chemically and electrolytically thinned.
50,000X.



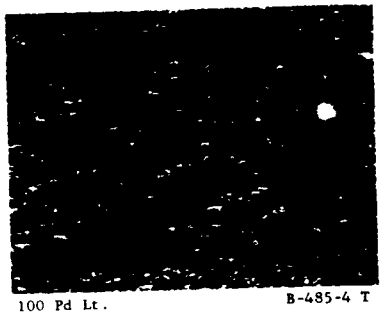
100X Pd Lt. B-485-3 T

(a) Transverse section

100X Pd Lt. B-485-3 L

(b) Longitudinal section

Fig. 18 - Microstructure of flat extruded from Brush QMV -200 mesh powder.



100 Pd Lt. B-485-4 T

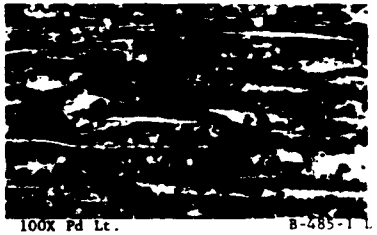
(a) Transverse section

100X Pd Lt. N-485-4 L

(b) Longitudinal section

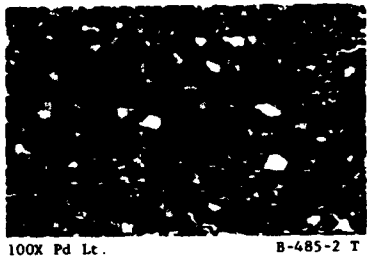
Fig. 19 - Microstructure of flat extruded from Pechiney SR -110 mesh powder

(a) Transverse section



(b) Longitudinal section

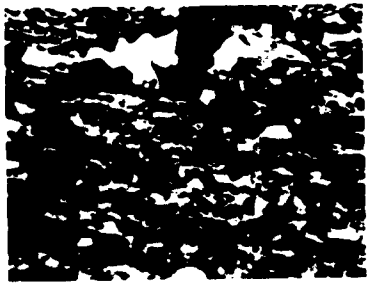
Fig. 20 - Microstructure of flat extruded from vacuum-melted Pechiney CR grade powder.



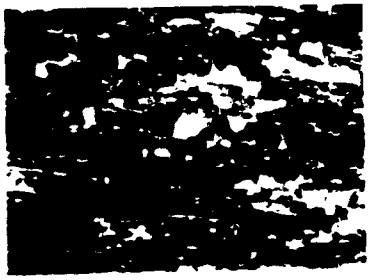
(a) Transverse section

(b) Longitudinal section

Fig. 21 - Microstructure of flat extruded from double-distilled beryllium powder.



(a) Transverse section 100X Pd Lt B 487a



(b) Longitudinal section 100X Pd Lt. B 487b

Fig. 22 - Microstructure of flat extruded from powder prepared from 5-pass zone-refined Pechiney CR beryllium.

the Brush QMV extrusion, Fig. 18, as a consequence of the low extrusion temperature employed (1650°F), had an unusually fine grain size of approximately 6 microns. The Pechiney SR powder extrusion, Fig. 19, with a larger initial particle size, had a grain size of approximately 10 microns. Examination of the longitudinal section of this extrusion, Fig. 19b, shows that a small amount of recrystallization has taken place, whereas no such behavior was exhibited by the Brush material. This is probably a consequence of a change in deformation and recrystallization behavior of beryllium with increasing purity.

The vacuum-melted Pechiney CR material, the double-distilled beryllium, and the zone-refined material were attritioned to an average particle size of 200 microns. Their grain sizes after extrusion consequently, were considerably higher, 20, 30 and 50 microns respectively. Note that while the Pechiney beryllium, Fig. 20b, was partially recrystallized, the double-distilled beryllium, Fig. 21b, and the zone-refined beryllium, Fig. 22b, appear to have undergone complete recrystallization.

The differences in microstructure described above can be attributed partly to differences in purity level and partly to differences in initial powder size. Differences in degree of preferred orientation probably exist also, and all of these effects must be considered in evaluating the mechanical properties of the various materials.

Chemical analyses of four of the extrusions are given in Table II.

Mechanical properties were evaluated, on all but the zone-refined material, by tensile and bend tests. Bend tests were performed on specimens 0.050-inch thick, and of varying widths up to 0.675 inch. Tests were performed on a single-point bending apparatus, employing a 0.2-inch radius ram. Bend angle at fracture as a function of specimen width for the various materials is shown in Fig. 23. The double-distilled material exhibited significantly greater transverse ductility than the other materials tested. The bend angles observed for the widest specimens

TABLE VII

Chemical Analyses of Beryllium Powder Extrusions

(in parts per million)

Material	Impurity			
	Fe	Ni	Al	Si
QMV	1115	171	540	238
Vacuum-Melted Pechiney CR	225	90	41	32
Pechiney SR	66	4	110	50
Double Distilled	16	1	18	20

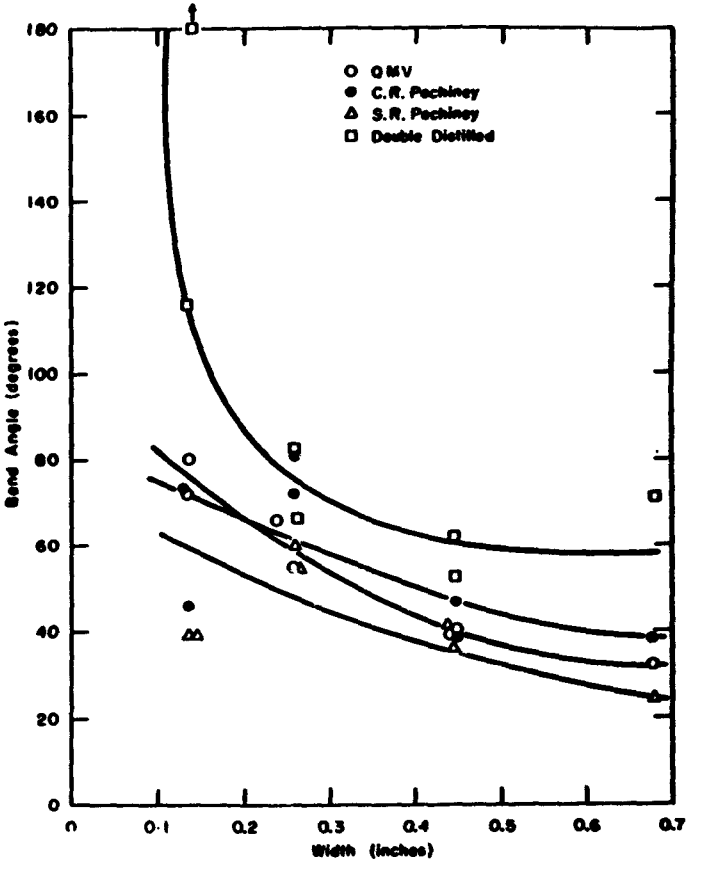


Fig. 23 - Bend ductility as a function of sample width for beryllium of different purity levels. Drawing No. RA-2367.

are higher (50 to 70°) than any reported for beryllium of comparable width to thickness ratios. Texture measurements are now being performed in order to determine if the high bend ductility is partly a consequence of the development of a favorable preferred orientation in high-purity beryllium under these conditions.

Tensile properties are presented in Table VIII. The high strengths reported for the QMV and SR extrusions appear to be a consequence of the extremely fine grain size of these materials. The low strength of the extrusion made from double-distilled beryllium is probably due both to the latter's larger grain size and to its higher purity. No large differences in tensile elongation were observed that might be attributed to purity, grain size or preferred orientation.

2. Oxygen Content of Distilled Beryllium

Samples of high-purity beryllium, prepared at NMI, have been analyzed by various techniques, including fast neutron activation, gamma activation and micro-vacuum fusion, by several laboratories, including Texas Nuclear Corporation, Austin, Texas, Atomic Weapons Research Establishment, Aldermaston, England, Atomic Energy Research Establishment, Harwell, England, and the Center for Nuclear Studies of Saclay, Gif-sur-Yvette, France.

Table IX is a summary of the results that have been obtained. It is difficult to draw definite conclusions from the data at this time, since different techniques were employed and samples of similar material did not necessarily come from identical pieces of material.

The wide range of values reported for vacuum-melted flake is somewhat disturbing, although, again, it should be pointed out that these samples were not all from the same billet. Perhaps more serious are the discrepancies among the analyses for zone-refined material, since these samples were taken from the same zone-refined bars.

The agreement for distilled material, on the other hand, is quite good, but this might be a fortuitous circumstance.

Arrangements are presently being made for an exchange of samples among the various laboratories in order to obtain additional data. Samples are also being analyzed by gamma activation at Lawrence Radiation Laboratories, Livermore, California.

TABLE VIII

Tensile Data on Powder Extrusions

Material	0.2% Offset Yield Strength (psi)	Ultimate Tensile Strength (psi)	Percent Elongation
QMV	71,400	111,000	7.3
Vacuum-Melted Pechiney CR	24,000	72,400	9.7
SR	61,400	95,600	5.4
Double-Distilled	22,500	63,700	7.6

TABLE IX

Summary of Oxygen Analyses on High-Purity Beryllium

Laboratory	Technique	Material	Oxygen (ppm)	Remarks
Aldermaston	Fast neutron	Vacuum-melted flake	55	Values observed for distilled Be depend on which side of sample faces target. Also, when sample surface is removed after irradiation and before counting, measured values apparently decrease by 30-40 ppm.
		Distilled	90-140	
		Distilled and Vacuum-melted	65- 90	
Texas Nuclear	Fast neutron	Vacuum-melted flake	302± 25	Results tentative until samples can be measured for fluorine by activation analysis, and until sample holders are rechecked.
		Distilled	80± 25	
Harwell	Micro-vacuum fusi...	Vacuum-melted flake	160	Reliability of method not well established at ≈60 ppm level.
		Distilled	60	
		1-pass zone refined	260	
		2-pass zone refined	300	
		3-pass zone refined	160	
Saclay	Gamma activation	Vacuum-melted flake	160± 50	Average of two determinations. Determinations not well defined, because of standard calibration and effect of surface contamination.
		Distilled	90± 40	
		1-pass zone refined	10± 5	
		2-pass zone refined	7± 5	
		3-pass zone refined	30± 10	

3. Future Work

The mechanical properties of double-distilled beryllium described above appear to be sufficiently promising to warrant more detailed evaluation. The powder metallurgy route for consolidation of distilled beryllium appears to be satisfactory in that it can yield samples having reasonably fine grain size. In future experiments it is planned to attrition double-distilled beryllium in a beryllium-lined ball mill. This procedure will permit the preparation of finer particle sizes, and will allow the process to be carried out under controlled atmosphere conditions. Sufficient double-distilled material has been generated to permit the study of several variables on mechanical properties, such as additional purification by zone refining, and the effect of texture.

A supply of Pechiney CR grade powder has been obtained for fabrication of the ball mill.

B. Fabrication and Evaluation of Fine-Grain Beryllium Produced from Ultra-Fine Powders - A. K. Wolff

1. Introduction

During the past quarter, work has continued on the chemical analysis of powder submitted by National Research Corporation and New England Materials Laboratory. The 150-ton inert atmosphere packing press has undergone extensive testing and revision, including the packing of two lots of Brush QMV powder to ensure proper procedures.

2. Analytical Results

The chemical analysis techniques employed during this period were carried out in the manner described in the previous quarterly report.[4] The results may be found in Section II, D of this report.

3. Packing Procedure

The basic unit of the packing press is shown schematically in Fig. 24. However, several additional features have been added to the setup. Since packing cans are frequently "frozen" in the die during

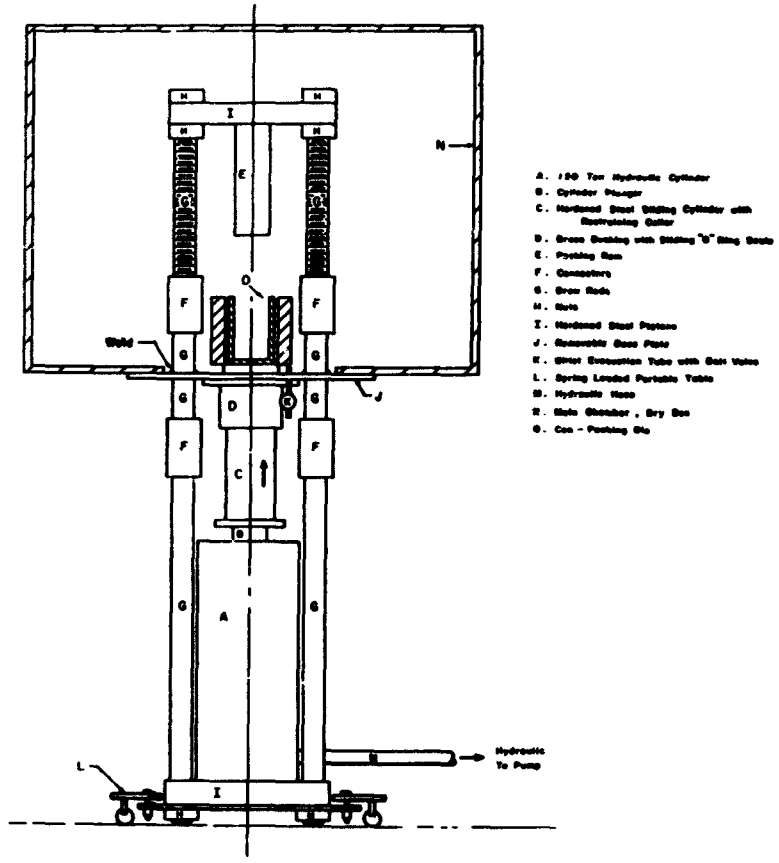


Fig. 24 - Schematic of inert-atmosphere press. Drawing No. RA-2366

pressing, several designs were tried which would utilize the 150-ton cylinder in removing the can from the die. Steel discs placed on the sliding cylinder and under the die walls were finally successfully employed to eject the can. A set of discs having varying heights have been machined for future use. A vacuum line has been installed on the small valve in the base of the drybox and will extend to the evacuation tube on the top of the billet, permitting evacuation of the billet in the drybox after welding. A filter consisting of spun glass fibers in a glass tube is located in the system to trap any residual powders which might be pulled out during evacuation. A water-cooled weld electrode was installed and checked for leaks under operating conditions. A special wrench was machined and will be used for assembling the press within the drybox.

Six cans (2.590-inch OD by 2.385-inch ID by 5.5-inch high) were machined and base plates welded to them. Six matching cover plates had evacuation tubes welded to them, and all welds were leak checked. A 2-5/8 inch ID packing die and a 2.365-inch OD packing ram were obtained and installed in the box. The relatively loose ram-to-can fit (approximately 0.010-inch clearance) was employed to prevent freezing of the ram after packing. The initial billet size is intended for powder lots of approximately 400 grams and is expected to yield compacts suitable for both hot pressing and extrusion with reductions of up to 25:1.

Packing procedures were tested using -200 mesh Brush QMV powder. The QMV powder was placed in glass jars 3-5/8 inch in diameter by 3-1/4 inch high, having a screw-type cover and sealed with electrical tape. All packing procedures were conducted under helium atmosphere. Several difficulties in the packing procedure became apparent during the first pressing operation, and these problems were successfully remedied for the second packing operation. The problems and solutions in order of occurrence were:

1. Pouring of the Powder.

It proved extremely difficult to remove powder from the wide-mouthed jars and place it in the mild steel can without considerable spillage. Neither pouring or spooning the powder was satisfactory,

since excessive agitation occurred in both cases. This problem would be magnified in the use of submicron or micron size particles. A special funnel-shaped pouring spout which screws directly onto the 3-5/8 inch diameter jar was made to facilitate the pouring operation. When powder is received, this cover can readily be exchanged for the original jar cover and pouring can then proceed with minimum agitation and spillage. A length of clear plastic tubing clamped to the top of the funnel permits great mobility of the pouring operation.

An attempt was made to enclose the pouring system completely by placing the can and glass container in a polyethylene bag, but this was found to be extremely awkward under the limited space conditions of the drybox. It is expected that small amounts of powder will certainly become atmosphere-borne during pouring and packing. However, the spilled powder can be slowly oxidized by the methods previously outlined for iron analysis, thereby eliminating the pyrophoricity problem. The toxicity problem can be handled by normal clean-up procedures.

b. Ejection of Powders During Insertion of the Ram.

As the ram was inserted into the can, gas pressure was built up ahead of it and powder was forced up the walls of the can and out into the drybox. Extremely slow packing speeds did not greatly alleviate this problem. A technique was devised that has been found to be completely successful in preventing ejection of the QMV powder and is expected to be similarly successful in the handling of submicron size powders. A standard "O"-ring, considerably smaller in diameter than the packing ram, is stretched over the ram. When the ram and can begin to overlap, the "O"-ring is brought down flush to the can top, and, as the ram continues to enter the can, the "O"-ring is automatically brought firmly into contact with the can top, thereby forming a powder-tight but not air-tight, seal. Initial attempts to use this technique were hampered by rolling of the "O"-ring, which tended to rotate 180 degrees at a time, thereby intermittently breaking the seal. However, it was found that, if the lip of the can was somewhat lower than the top of the packing die, the "O"-ring would be forced into the space between the die and ram, thereby preventing rolling.

c. Handling of the Die and Can.

The packing die was found to be extremely heavy and difficult to handle during ejection of the can from the die after packing. During the early procedural checkouts, the gloves were pinched under the die and cut, and had to be replaced. There was also the potential hazard of breaking of windows or fixtures inside the drybox. The handling difficulty was overcome by the design of a special support mounted on one of the draw rods in such a way that it could be adjusted to hold the die at any desired height. This procedure minimized lifting of the die and also eliminated the possibility of splitting of the can walls during repositioning of the die.

d. Drawing of Beryllium Powder into the Weld Zone.

It was found that, during the heliarc welding of the billet, residual beryllium powders were drawn up the walls of the can and into the weld zone, resulting in a porous weld. Brushing off the powders adhering to the can walls did not alleviate this problem and, because of expansion of the can during welding, the use of close tolerance covers was not practical. This problem was overcome by use of a dummy cover, approximately 1/8-inch thick and tapering from a maximum diameter of 2.400 inches to a minimum diameter of 2.385 inches. After packing of the powders, this dummy cover (which contains a hole matching the evacuation hole on the actual cover) is positioned on top of the can with the minimum diameter downward and pressed into the can with the packing ram at a pressure of approximately 5,000 psi. This serves the double purpose of forcing down into the can any powders adhering to the walls and of preventing loose powders from rising up the walls during welding.

e. Location of Weld Leaks.

After the first QMV powder compact was made, the cover was welded to the can in the drybox under helium. Difficulty in evacuating the can indicated that leaks were present in the weld. Since volatile contaminants could not be introduced into the box, and since

the can is evacuated against one atmosphere of helium, standard leak testing techniques were not possible. It was found that the careful application of Apiezon sealing compound, a putty-like material, could successfully pinpoint the leak. The compound was applied to the suspected area while pumping on the billet, and the vacuum gauge responded rapidly to the application or removal of the Apiezon over the leak. The area could then be rewelded and the procedure repeated.

f. Crimping of the Evacuation Hose.

After evacuation of the billet the evacuation tube was flattened under the packing ram while pumping on the billet. This was intended to produce a nearly leak-tight seal which could then be heated with the welding torch to produce a vacuum-tight can. It was found during the course of the compacting of QMV powder that pressures necessary to sufficiently flatten the evacuation tube resulted in cracking of the tube, thereby preventing adequate evacuation of the billet. Several subsequent tests were made involving various packing pressures, ram geometries, and evacuation tube materials. It was found that the problem could be overcome by the use of 304 stainless steel instead of mild steel for evacuation tubes. Packing pressures of about 30 tons over tubing lengths of approximately 2-1/2 inches achieved a leak-free crimp which was successfully sealed by welding.

It is felt that techniques are now sufficiently developed for the packing of ultrafine powders submitted by NRC and NRM Lab. In anticipation of these operations, the drybox has been thoroughly cleaned and all tubing in the helium inlet system and the inert atmosphere purification system, plus all the drying elements, have been replaced. All glass parts of the system have been removed, cleaned and regreased preparatory to beginning the pressing operations.

IV. REFERENCES

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